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Rubber – Determination of metal content by atomic absorption spectrometry – Part 2: Determination of lead content

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Gummi – Bestämning av metallhalt genom atomabsorptionsspektrometri – Del 2: Bestämning av blyhalt

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ISO 6101-2:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6101-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 6101-2:1986), which has been technically revised.

ISO 6101 consists of the following parts, under the general title *Rubber — Determination of metal content by atomic absorption spectrometry*:

- *Part 1: Determination of zinc content*
- *Part 2: Determination of lead content*
- *Part 3: Determination of copper content*
- *Part 4: Determination of manganese content*
- *Part 5: Determination of iron content*

Annex A forms an integral part of this part of ISO 6101.

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Rubber — Determination of metal content by atomic absorption spectrometry —

Part 2: Determination of lead content

WARNING — Persons using this part of ISO 6101 should be familiar with normal laboratory practice. This part of ISO 6101 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 6101 specifies an atomic absorption spectrometric method for the determination of the lead content of rubbers.

The method is applicable to raw rubber and rubber products. There is no limit to the concentration of lead that can be determined. High or low concentrations may be determined, provided that suitable adjustments are made to the mass of the test portion and/or the concentration of the solutions used. The use of the standard-additions method may lower the bottom limit of detection.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 6101. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 6101 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling.*

ISO 247:1990, *Rubber — Determination of ash.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1772:1975, *Laboratory crucibles in porcelain and silica.*

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

3 Principle

If the rubber does not contain halogenated compounds, a test portion is directly ashed at $550\text{ °C} \pm 25\text{ °C}$ as specified in ISO 247:1990, method A. If the rubber contains halogenated compounds, a test portion is first mineralized with sulfuric and nitric acids, the acids are removed by evaporation, and the portion is ashed at $550\text{ °C} \pm 25\text{ °C}$.

NOTE — The presence of even small amounts of halogens may lead to the loss of volatile lead salts during dry ashing.

The ash obtained is boiled with ammonium acetate solution to dissolve the lead. Insoluble lead silicates, if present, are converted to chloride by boiling with a mixture of hydrochloric acid, nitric acid and hydrogen peroxide.

The solution is aspirated into an atomic absorption spectrometer and the absorbance is measured at a wavelength of 283,3 nm, using a lead hollow-cathode lamp as the lead emission source.

NOTE — ISO 6955:1982, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*, defines the spectrometric terms used in this part of ISO 6101.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

4.1 Sulfuric acid, $\rho_{20} = 1,84\text{ Mg/m}^3$.

4.2 Nitric acid, $\rho_{20} = 1,40\text{ Mg/m}^3$.

4.3 Hydrochloric acid, $\rho_{20} = 1,18\text{ Mg/m}^3$.

4.4 Hydrochloric acid, diluted 1 + 2.

Dilute 1 volume of concentrated hydrochloric acid (4.3) with 2 volumes of water.

4.5 Hydrogen peroxide, 30 % (m/m) solution.

4.6 Ammonium acetate, 180 g/dm³ solution.

4.7 Standard lead stock solution, containing 1 g of Pb per cubic decimetre.

Either use a commercially available standard lead solution, or prepare as follows:

Weigh, to the nearest 0,1 mg, 1 g of metallic lead (purity $\geq 99,95\%$) and transfer to a 100 cm³ beaker (5.12). Add 30 cm³ of water and 20 cm³ of nitric acid (4.2) and boil on a sand-bath (5.10). If the lead is dissolved, continue boiling until the solution is reduced to about 20 cm³ or less. No nitrogen oxides should be observed. Otherwise, add water and continue boiling. Transfer to a 1 000 cm³ one-mark volumetric flask (see 5.7) with 1 + 2 hydrochloric acid (4.4) and fill to the mark with 1 + 2 hydrochloric acid (4.4).

1 cm³ of this standard stock solution contains 1 mg of Pb.

4.8 Standard lead solution, containing 10 mg of Pb per cubic decimetre.

Carefully pipette 10 cm³ of the standard lead stock solution (4.7) into a 1 000 cm³ one-mark volumetric flask (see 5.7) dilute to the mark with 1 + 2 hydrochloric acid (4.4), and mix thoroughly.

Prepare this solution on the day of use.

1 cm³ of this standard stock solution contains 10 µg of Pb.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Atomic absorption spectrometer, fitted with a burner fed with acetylene and air, compressed to at least 60 kPa and 300 kPa, respectively, and also fitted with a lead hollow-cathode lamp as the lead emission source. The instrument shall be operated in accordance with the manufacturer's instructions for optimum performance.

Alternatively, an **electrothermal atomization device (graphite furnace)** may be used. It shall be operated by a competent person in accordance with the manufacturer's instructions for optimum performance, during drying, ashing and volatilization of the test portion.

5.2 Balance, accurate to 0,1 mg.

5.3 Muffle furnace, capable of being maintained at $550\text{ °C} \pm 25\text{ °C}$.

5.4 Evaporating dish, of capacity about 100 cm³, made of quartz glass, with a clock-glass cover.

5.5 Conical flask, of capacity 250 cm³, made of silica or borosilicate glass.

5.6 Measuring cylinder, of capacity 25 cm³, graduated in 0,5 cm³ divisions.

5.7 One-mark volumetric flasks, glass-stoppered, of capacities 50 cm³, 100 cm³, 200 cm³, 500 cm³ and 1 000 cm³, complying with the requirements of ISO 1042, class A.

5.8 Filter funnel, 65 mm diameter, 60° angle.

5.9 Volumetric pipettes, of capacities 5 cm³, 10 cm³, 20 cm³ and 50 cm³, complying with the requirements of ISO 648, class A.

5.10 Electric hotplate or heated sand bath.

5.11 Steam bath.

5.12 Beaker, of capacity 100 cm³.

5.13 Crucible, of platinum, and of capacity 50 cm³ to 150 cm³ depending on the test portion size.

5.14 Crucible, of silica or borosilicate glass, of capacity 50 cm³ to 150 cm³ depending on the test portion size, complying with the requirements of ISO 1772.

6 Sampling

Carry out sampling as follows:

raw rubber: in accordance with ISO 1795;

latex: in accordance with ISO 123;

products: to be representative of the whole batch.

7 Procedure

7.1 Test portion

7.1.1 If the rubber does not contain halogenated compounds, weigh, to the nearest 0,1 mg, 0,5 g to 2,0 g (depending on the expected lead content) of milled or finely cut rubber into a crucible (5.13 or 5.14).

NOTE — It is advisable to run the Beilstein test on a preliminary test portion for halogen content, as volatile lead compounds are possibly formed in the presence of halogen-containing admixtures.

7.1.2 If the rubber contains halogenated compounds (for example, in the case of chloroprene rubber), weigh, to the nearest 0,1 mg, 0,5 g to 2,0 g (depending on the expected lead content) of milled or finely cut rubber into the conical flask (5.5).

7.2 Preparation of test solution

7.2.1 Destruction of organic matter

7.2.1.1 If the rubber does not contain halogenated compounds, ash the test portion in accordance with method A of ISO 247:1990.

7.2.1.2 If the rubber contains halogenated compounds (7.1.2), use the following alternative procedure:

To the flask containing the test portion, add 10 cm³ to 15 cm³ of sulfuric acid (4.1) and heat moderately on the electric hotplate or on the sand bath over a gas burner (5.10) until the rubber has disintegrated. Carefully add 5 cm³ of nitric acid (4.2). Continue heating until the rubber has completely decomposed and white fumes are evolved.

Some rubber formulations cause considerable splashing; in this case, use a larger conical flask.

Transfer the reaction mixture quantitatively into the evaporating dish (5.4), evaporate to dryness and ash in the muffle furnace (5.3), maintained at 550 °C ± 25 °C, until all the carbon has burned off.

7.2.2 Dissolution of inorganic residue

After cooling, boil the residue obtained in 7.2.1.1 or 7.2.1.2 with 15 cm³ of ammonium acetate solution (4.6) and filter through a paper filter. Retain this filtrate (A) and ash the filter with the residue in the muffle furnace (5.3), maintained at 550 °C ± 25 °C, until all the carbon has burned off.

Cool to room temperature, add a mixture of 10 cm³ of hydrochloric acid (4.3), 5 cm³ of nitric acid (4.2) and 10 drops of hydrogen peroxide (4.5), then boil for about 10 min.

Cool and filter the reaction mixture. Evaporate the filtrate on the steam bath (5.11) to dryness and take up the residue with water. Add this to filtrate A. Transfer the combined solutions, quantitatively, to a 50 cm³ one-mark volumetric flask (see 5.7) with hydrochloric acid (4.4). Dilute to the mark with hydrochloric acid (4.4) and proceed in accordance with 7.4.

Test solutions in hydrochloric acid shall be of the same concentration as in 4.4. If evaporation, etc., has reduced or increased this concentration, adjust it accordingly with concentrated hydrochloric acid (4.3) or with water.

7.3 Preparation of the calibration graph

7.3.1 Preparation of calibration solutions

7.3.1.1 Into a series of five 100 cm³ one-mark volumetric flasks (see 5.7) introduce, using pipettes (see 5.9), the volumes of standard lead solution (4.8) indicated in table 1. Dilute to the mark with 1+2 hydrochloric acid (4.4) and mix thoroughly.